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CONFIDENTIAL**THEORY OF THE PRODUCTION OF ARTIFICIAL GRAPHITE BY
THE GRAPHITIZATION OF CARBONACEOUS MATERIALS**

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Introduction

The industrial production of artificial graphite was worked out by the American engineer Acheson at the beginning of the 1890's. The process consists of heating a carbonaceous material to an extremely high temperature (more than 2200°) in an electrical resistance furnace. After this treatment the carbonaceous material becomes greasy to the touch, acquires the ability to write on paper, its electrical conductivity and chemical stability are greatly increased, etc.: in a word, it acquires the properties characteristic for natural graphites. Chemical analysis of graphitized carbonaceous materials shows that they consist almost entirely of carbon and contain a negligible quantity of foreign substances: 0.1-0.1% of mineral impurities and 0.1-0.3% of adsorbed volatile substances. This alteration of the properties of a carbonaceous material is designated by the technical term "graphitization" of the material.

In spite of the fact that in nearly all leading countries there are powerful industrial installations for the production of artificial graphite, the theory of this process has not been worked out at all, which has created favorable conditions for keeping the production in secret. Therefore, when we had to take part in mastering this production in the [Soviet] Union, we ran into great technical difficulties which required us to engage in studies of the physico-chemical fundamentals of the process and the methods of its control as well as in technological research.

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According to modern ideas, the chief component part of carbonaceous materials is graphite, which is found in a highly dispersed state. Besides graphite there usually enter into the composition of carbonaceous materials in the form of "admixtures" (i.e., uncharacteristic component parts) highly polymerized compounds of carbon, different inorganic substances, and adsorbed matter.

From this point of view carbonaceous materials must be considered as polycrystalline, highly dispersed substances, and it must be expected that their conversion on heating into "artificial graphite" will proceed in compliance with the laws of the thermal recrystallization of polycrystalline substances ⁽¹⁾.

We used these general concepts as a basis of work on the theory of the production of "artificial graphite". Here we set the technical term "artificial graphite" in quotes to emphasize its incompatibility with the theoretical positions just stated. This term was introduced at a time when amorphous carbon was considered an independent allotropic form and the basic constituent of carbonaceous materials. From this point of view a carbonaceous material, when heated to a sufficiently high temperature, was converted into graphite, i.e., graphitized. Now, when carbonaceous materials are considered as highly dispersed graphite, this terminology does not correspond to the essence of the matter; nevertheless, it is generally accepted, and with the above-stated reservations we will use it.

It is well known that different carbonaceous materials do not lend ~~them~~ ^{to graphitization} themselves with equal facility and yield products which have very different properties. The explanation of such characteristics is completely impossible without the development of a general theory of graphitization.

Thus in essence graphitization boils down to an alteration of the structure of a carbonaceous material with respect to dispersion.

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Every type of surface existing in a dispersion system has its own excess of specific free surface energy (σ_1). So that, if the surface energies of the different types of surfaces are $S_1\sigma_1$, $S_2\sigma_2$, $S_3\sigma_3$, etc., then the total surface energy of the system is

$$S = S_1\sigma_1 + S_2\sigma_2 + S_3\sigma_3 + \dots = \sum S_i\sigma_i.$$

The higher the dispersion, the greater the magnitude of the total surface (S); the more developed are the surfaces with a large specific energy, the greater is the average specific surface energy (σ). In this consists the connection between the dispersion state of the system and its store of free energy, which determines its propensity towards recrystallization. Obviously the measure of this propensity will be the concentration of surface energy

$$\frac{S\sigma}{V} = \frac{S_1\sigma_1}{V} + \frac{S_2\sigma_2}{V} + \dots = \sum \frac{S_i\sigma_i}{V}.$$

However, it is not in all disperse systems that the tendency towards alteration of the dispersion state can easily express itself. Thus in crystalline agglomerates at low temperatures we discover no alterations; only on heating to a definite temperature - characteristic for the given substance - does recrystallization begin: i.e., in these systems something hinders the completion of the processes which lead to reduction of the free energy; they are stabilized systems similar to stabilized colloidal solutions.

On the basis of all experimental data obtained up to this time it must be assumed that the displacement of the structural elements of crystals can take place in the solid phase, and obviously, only under conditions of the immediate contiguity of the crystals. A search also should be made for the cause of the stability under conditions of contact of the crystalline agglomerates at low temperatures.

Recrystallization at every given temperature proceeds only to a fixed limit; the temperature determines the degree of coarsening of the

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crystalline grain, and cannot be compensated for by the duration of heating. From this it follows that the conditions of contact of crystalline grains change with the temperature.

Thus the ability of carbonaceous materials to be graphitized is a function of their dispersion state, since this state determines the conditions of the contiguity of crystal grains. (By the term dispersion state literally structure we indicate the whole complex of phenomena which characterize the structure of the substance out of parts which are separated one from the other by boundary layers whose thickness is equal to or greater than that of the boundaries between phases.)

The published material relative to the capacity of different carbonaceous materials to undergo graphitization has been very meager and contradictory. Fitzgerald ⁽²⁾ states that the best graphite is produced from anthracite and attributes this to the presence in it of catalytic mineral admixtures. Arsem ⁽³⁾ arranges carbonaceous materials in the following order of increasing facility of graphitization: lamp-black, anthracite, foundry coke, and petroleum coke. This order agrees with Ruff's ⁽⁴⁾ data: charcoal, flame, carbonblack, anthracite, ash cokes, and cokes with a low ash content. But Arndt gives another order ⁽⁵⁾: charcoal, new anthracite, gas coke, old anthracite, petroleum coke, foundry coke, and retort carbon. This exhausts the literary data. They contradict not only one another, but also our observations. This results from the differences in meaning which the authors invested in the term "graphitization", and from the inadequacy of description of the coals they investigated.

Our observations, it appears, will be able to bring about some clarification.

Methods of Work

We carried out the heating of carbonaceous materials in a furnace of very simple construction (Figure 1). It had already been used by us

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for studying the thermic treatment of natural graphites in 1930⁽⁶⁾, and for work on graphitization it had only to be adapted for measurement of the temperature. The furnace consists of a carbon tube (P) held between two large carbon electrodes (ee) through which a low voltage heating current is conducted. To protect it against combustion and ~~to reduce~~ ^{to reduce} the heat emission, the tube is surrounded on all sides by powdered charcoal (c). Since the temperature of the tube near the electrodes is considerably lower than at its mid-point as a result of increased heat emission in these places, plugs of charcoal (dd) are inserted at both ends of the tube. This prevents the material being treated from coming into contact with the electrodes and assures its more even heating. For temperature measurement another tube (T) was placed in the furnace perpendicular to the working tube, which was in the horizontal position. An optical pyrometer of the Holborn-Kurlbaum system with an incandescent lamp was sighted on the working tube through tube T. Temperatures measured in this manner can make no pretense to absolute value, or even to great accuracy, since the smoke issuing from the furnace screens the glowing surface. However, after some practice one can succeed in carrying out measurements with sufficient accuracy to establish the general relationships of the graphitization process. For the average temperatures of our experiments at ~2200° we rate the accuracy of measurements as $\pm 25^\circ$. The temperatures in all summaries of the results of this work are given as "black", i.e., without corrections for the reduced light emission of the carbon tubes.

A large obstacle to the study of the thermic treatment of carbonaceous materials is the absence of direct methods for measuring the degree of graphitization; only indirect methods are known for this, such as measurements of electrical conductivity, combustibility, density, ash content, x-ray measurements, etc. All of these permit conjectures on the alteration of the dispersion of the material being treated, but they do not provide an absolute measure of the degree of dispersion.

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The most noteworthy among these techniques is the x-ray method, which promises to become the most perfect method for the investigation of dispersion states, since it permits determination of the spatial distribution and the form of the individual crystal grains which compose the substance (7). However, great difficulties are connected with x-ray investigation of dispersoids due to the complexity of the apparatus and the insufficient development of measurement techniques. Therefore, in the present work we employed this method only for the qualitative comparison of the structures of different carbonaceous materials. A future article will be devoted to systematic quantitative research on the dispersions structures of graphitic substances, but this work has not yet been concluded. Besides, the x-ray diagrams taken by K. V. Vasil'yev (Figures 2 and 3) show so graphically the differences in structure of carbonaceous materials that even the mere qualitative comparison of them permits extremely important conclusions to be reached. Thus it was on the basis of these results that we succeeded in establishing the connection between the capacity for graphitization and the orientation of the crystal grains in the carbonaceous material.

In Figure 2 is shown a typical Debye-Scherrer diagram of a highly-dispersed carbonaceous material which was obtained at a low temperature. The interference rings of graphite here are hardly discernible; the thickness of the crystals was here estimated at approximately 10-20 Å. Figure 3 gives the x-ray diagram of a graphitic substance with an oriented distribution of crystals.

Other methods for detecting alterations of the dispersion structures lead to results which are less easily visualized, but then they possess the great advantage of simplicity of execution. Besides, they are all closely connected with the technically important properties of carbonaceous materials. In graphitization it is most convenient to study the electrical conductivity, combustibility, and density of the carbonaceous materials.

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We carried out the determination of the electrical conductivity of powders according to the Arndt method under a pressure of 110-120 kg/sq cm ⁽⁸⁾. Unfortunately, this property in the case of highly dispersed substances depends on such a large number of factors that the result is not susceptible to quantitative treatment. There is no doubt that conductivity depends strongly on the size of the crystal grains and can therefore serve as a relative measure of their growth. The accuracy of Arndt's method in the best case is $\pm 5\%$.

The determination of combustibility was carried out by the method which we developed for evaluating natural graphites ⁽⁹⁾. In it a weighed portion of the powder (1 g) is placed in a flat porcelain boat (5 X 3.5 X 1 cm) in a tubular furnace ($\phi = 5$ cm) placed on an incline (3-5°) and open at both ends, and first heated to a definite temperature; when 10 minutes have passed, the boat is taken out, cooled in a desiccator, and weighed; then the experiment is repeated with the same weighed portion at a higher temperature. The temperature at which 3% of the weighed portion burns off is accepted arbitrarily as the measure of combustibility. The accuracy of its determination is rated at $\pm 10^\circ$.

The rate of combustion of graphite at a constant temperature $\left(\frac{\partial m}{\partial t}\right)_{T,C}$ is determined by the rate of diffusion of gases, the rate of reaction, and the size of the active surface (S_a). For the stationary state of the process, it can be assumed that the composition and constitution of the gaseous phase are constant. Then $\left(\frac{\partial m}{\partial t}\right)_{T,C} = K S_a$, where K is the constant of the rate of combustion which does not depend on the dispersion structure of the graphite; $S_a = S \cdot A$, i.e., the active surface (S_a) is equal to the external geometric surface (S) multiplied by the activity of this surface (A). A is the measure of the surface concentration of active atoms of carbon and stands in direct connection with the dispersion of the burning graphite substance.

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Density is an extremely important characteristic of highly dispersed substances, but it changes little on account of graphitization; therefore it must be measured by the most accurate methods, and this involves considerable difficulties. Besides the rule for variation of density is very different for different carbonaceous materials. Therefore, measurement of density has had limited use in the study of graphitization. We determined the density pycnometrically in xylene with an accuracy of ± 0.003 .

All these composite properties of crystalline agglomerates vary in general uniformly during graphitization and tend toward a "graphite" limit. The general form of the dependency of these properties on the temperature of heating is shown in Figure 4. For every property there is a temperature interval within the limits of which it the property changes abruptly. Ordinarily, the greater this interval, the higher the temperature of the point of change of curvature (OA). The point of change of curvature, corresponding to the greatest change in the property, and the temperature coincident with it are characteristic for each carbonaceous material.

When a carbonaceous material produced at a low temperature is heated, the first sign of structural change, as Ruff ⁽⁴⁾ showed, is the loss of its sorption activity and its capacity to be activated. Abrupt changes in this property take place between 1100 and 1400°. At higher temperature^s other properties begin to be altered. Moreover, for different carbonaceous materials the temperatures of abrupt changes can differ greatly. Thus, the combustibility of cokes falls sharply between 1400 and 1800°, but that of anthracites between 1900 and 2000°. The electrical conductivity^t of very different carbonaceous materials increases^{sharply} between 1900 and 2000°; retort carbon and carbon black are the exceptions; the first gives a point of change of curvature at a considerably higher temperature, while carbon black generally changes its properties only

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insignificantly at the temperatures of our experiments (Table 6). In general the most abrupt alteration in the majority of properties is observed during the transition of the carbonaceous material from the region of colloidal dispersion to the region of coarsely dispersed substances. It is precisely this change which constitutes graphitization. The temperature of this transition can be very different for different carbonaceous materials.

Easily graphitizable carbonaceous materials complete this transition at about 2000°, and the rate of further change of properties slows down; charcoal is graphitized at about 2500°, while carbon black hardly shows a sign of graphitization even at 2700°.

Results

Experiments on tar coke, charcoal, and carbon black have shown that, on heating, the "graphitization equilibrium" is established very quickly, so that the duration of heating and the period of ~~bringing~~ bringing the material to a constant temperature have no noticeable influence.

The results of the basic experiments are given in Tables 1-8 and shown graphically in Figures 6-12.

Tar Coke (Table 1, Figure 5) is a product of the destructive distillation of coal tar; it was prepared in the factory "Electrougli"; it was roasted at 1400° and pulverized until it would pass through a 140-mesh screen (i.e., 140 openings to the inch); moisture content was 2.05%; volatile components (by the crucible test) comprised 3.29%.

Petroleum Coke (Table 2) is a product of the destructive distillation of the petroleum residues at the factory imeni Budennyi (Baku); it was roasted at 1400° and pulverized until it would pass through a 130-mesh screen; moisture content was 0.32%; content of volatile substances was 0.95%.

Coal Coke (Foundry) (Table 3) was sifted through a 50-mesh screen: Analysis of the ash of coal coke in percent:

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SiO ₂	48.54
Al ₂ O ₃	4.66
Fe ₂ O ₃	35.90
CaO	6.22
MgO	0.23

The graphitization products of these three cokes are similar to each other both as to properties and external appearance. They are all solid and hard to the touch, write well on paper, and have a sandy consistency. Graphitized coal coke is somewhat greasier than the others, which apparently results from the higher ash content of the original material.

Charcoal (birch) is characterized in Table 4. It was roasted at 700° and pulverized until it would pass through a 130-mesh screen; moisture content was 3.86%; content of volatile substances was 11.72%.

Graphitized charcoal differs considerably from the products of the graphitization of cokes; after graphitization it becomes softer, but is still harder than T-cokes.

Retort Carbon (Table 5) is the product of the pyrogenous decomposition of gaseous carbon compounds at the Moscow Gas Factory. It was roasted at 1400° and pulverized until it would pass through a 130-mesh screen; the moisture content was 0.23%; content of volatile substances was 0.39%; density was 2.115.

After graphitization retort carbon becomes considerably softer and writes well on paper; its external form is not changed, but it becomes silvery and softer than all the other T-products.

Flame Carbon Black No. 3 (Table 6) is derived from petroleum residue of the Kudinov Carbon Black Factory. Ash content was 0.12%; moisture content was 0.92%; volatile content was 0.68%. When graphitized, carbon black remains almost completely unchanged in external form and properties. Only in x-ray diagrams can a certain decrease in dispersion be discovered.

Donets Anthracite (Table 7) comes from the mine imeni Voykov of the Sverdlovsk Mine Administration. (Stratum 2/5 Ku). Moisture content was 1.90%.

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Poltavka Anthracite (Table 8) comes from mine No. 4 near the village of Poltavka in the Southern Urals. Moisture content was 7.12%; volatile content was 7.67; sulfur (S) content was 0.22%.

Both of these anthracites yield a practically identical graphitization product, which is totally unlike the other graphitized carbonaceous materials. It has a flaky texture, is very greasy and soft to the touch, and resembles natural, well-crystallized graphites. This circumstance deserves particular attention, because the chemical composition of these anthracites is very different. The Donets anthracite contains a large quantity of organic compounds, and in the ash part iron and calcium. The Poltavka anthracite contains almost none of these admixtures. These anthracites are just as clearly distinguishable according to their macroscopic structure; the Poltavka type has a thin-layered structure, while the Donets type has a completely dense structure in small pieces and exhibits conchoidal fracture.

Analyses of the ashes (in percent):

	<u>Donets</u>	<u>Poltavka</u>
SiO	32.56	69.42
Al ₂ O ₃ }	41.81	23.39
Fe ₂ O ₃ }		
CaO	15.81	0.74-3.79
MgO	1.93	0.29

In view of the fact that there are in literature definite indications pointing to the catalytic action of substances which are capable of forming carbides, it was necessary to study this factor. Thus the admixture of SiO₂ was for a long time considered necessary for the production of good artificial graphite (2, 10, 11, 12, 13). But the investigations which have been carried out have shed little light on the subject, since their results contradict one another.

Ruff (4) gives the following series of catalysts for the graphitization of tar carbon black which has been briquetted with 30% of tar: B, Al, B₂O₃, Al₂O₃, Fe, Mn, Si, SiO₂, MgO, BaO, CaO; Bi and Sn do not catalyze at all; the oxides show less effect than the elementary substances.

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Schlaifstein ⁽¹⁴⁾ gives another series of catalysts for petroleum coke: B, Cr, Fe, Ni, Al, Mn, Mg, Ca; Si has absolutely no effect according to him.

However, these data do not make it possible to evaluate the practical importance of inorganic admixtures. Therefore, in order to investigate this factor, we conducted a series of experiments with petroleum coke to which were added powdered CaO , B_2O_3 , Fe_2O_3 , Al_2O_3 , and SiO_2 , and the mixture as usual was placed in the carbon tube. The results of these experiments are cited in Table 9. From these results it is evident that the above-mentioned substances exert only a minor effect. Although the graphitization product obtained with the admixtures was a little softer and more greasy to the touch than that obtained without them, there is no doubt but that for the graphitization of powders the presence of these substances cannot have any practical value.

However, it has been definitely established that certain anthracites (Tables 7 and 8) yield graphitization products of a completely different type than all the other carbonaceous materials. This is impossible to explain on the basis of the composition of their ashes because, on one hand, they have ashes of very different composition, and, on the other hand, coal coke (Table 3) which has almost the same kind of ash as Donets anthracite yields a graphitization product of an entirely different type.

Thus the widespread opinion in literature of the strong effect of admixtures which form carbides on graphitization does not correspond to reality. This opinion has been based apparently on the observation of the formation of flakes of graphite during the decomposition of carbides. This phenomenon should not be confused with the process of graphitization in the narrow sense of the word; the latter proceeds in a solid phase of constant composition and obeys the law of thermal recrystallization of polycrystalline substances ⁽¹⁾. But the formation of flakes of graphite during the decomposition of carbides takes place according to the law of crystallization from solutions on evaporation

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of the solvent; the solution can be either liquid or solid; it has a complex changing composition and consists of carbides and the products of their dissociation.

In those cases when any considerable quantity of inorganic substances get into a factory furnace filled with carbonaceous powder, it is possible to observe the formation of drops of fused graphite. These drops have an interesting flaky structure (Figure 12) and often contain empty inside spaces which are partially filled with thin leaves of graphite. In purely carbonaceous powders such drops are never observed, no matter how high the temperature. This phenomenon is explained by the fact that carbides when dissolved in graphite lower its melting point. On further heating the carbide-forming elements are evaporated and the graphite crystallizes out of the solution.

To produce coarse graphite crystals we placed large pieces of iron in the factory furnace. In this case it seems that the following processes take place: the iron is melted and is saturated with carbon in proportion as the temperature is increased; when the temperature is increased so much that the iron begins to evaporate, the graphite gradually crystallizes out and, if the furnace attains a high enough temperature, then crystals of almost pure graphite remain in it. By evaporating such iron solutions we succeeded in obtaining crystals of graphite up to 2 cm in length (Figure 13).

The evaporation of different admixtures on heating is pictured in Figure 14. Calcium evaporates easier than the others, and then silicon and aluminum (2200°); boron and iron hold on a little more firmly.

All the admixtures which we have studied are evaporated at 2500°, leaving only a small residue which is determined by the concentration of the vapor.

A sharp decrease of the ash content of industrial materials having a high ash content takes place between 1800 and 2000°.

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Examining the experimental data we can arrive at a conclusion which fully corresponds to the theoretical concepts. In particular, the reason for the differing behavior of carbonaceous materials when graphitized is due to their dispersion structure, and not to their chemical composition. Thus, there is no disagreement relative to the difficulty of graphitizing carbonaceous materials of porous structure. Inversely, the more dense and solid is the carbonaceous material, the easier it can be graphitized.

Furthermore, the graphitization products of different carbonaceous materials possess such different properties that one obviously should not place them in the same category. Obviously not only the density of the structure of the carbonaceous material, but also the mutual arrangement of the graphite crystallites which compose the carbonaceous material and the conditions of their contact determine its behavior on heating.

Among the carbonaceous materials investigated, it is possible to distinguish between the following types of dispersion structures:

1. Flaky structure (carbon black, soot, smoke, dust, etc.). From the point of view of formation these are products of the coagulation of ashes. From the structural point of view they consist of porous concretions of variously dimensioned graphite crystals combined into flakes. In them the conditions of contact between crystals are especially unfavorable for recrystallization, and therefore, they are practically not graphitized.

2. Thin layer structure (carbonaceous materials derived from plants, and animal fermoids carbonaceous materials of animal origin). Characteristic of these is the partial preservation of the structure of the original organisms. This causes the formation of their thin layer structure; typical representatives of this type have a layer thickness of the order of colloidal dimensions. As a result of this they undergo graphitization with difficulty.

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3. Dense or compact crystalline structure (shiny, carbonaceous materials Glanzkohlen for example retort carbon). They are characterized by extremely dense packing of crystallites and great hardness. This structure favors thermal recrystallization; these materials are easily graphitized.

4. Foamy structures (coke in the narrow sense afroids). As far as their origin is concerned, these are products of the carbonization of semi-liquid masses. They consist of bulky layers, distinguishable by the naked eye. They are easily graphitized.

5. Crystalline layer structures (certain coals). These should be considered as substances formed while the material is in a state of flux (fluids). This explains their highly oriented crystalline structure. The high orientation of the crystallites and their dense structure are extremely favorable to graphitization; they yield shiny, unusually greasy artificial graphite, suggesting natural flaky graphites. No other carbonaceous materials yield such a graphitization product.

The ability of certain anthracites to yield an artificial graphite of this type was earlier attributed to the presence in them of mineral admixtures. Our x-ray investigations have shown that this property is possessed only by those carbonaceous materials in which a high orientation of the graphite crystallites occurs; these crystallites are so disposed that their basic plane coincides with the plane of stratification of the texture of the coal. This orientation is also preserved in the graphitization product, which is the cause of its flaky structure and high greasiness.

6. Layer structures formed by crystals not highly oriented (the majority of coals and cryptocrystalline graphites). In relation to graphitization they occupy the mid-point between the shiny carbonaceous materials and the cokes. The coals of the Moscow Basin and the Kureisk and Noginsk graphites belong to this group.

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Results and Conclusions

1. Comparative studies of the graphitization of different carbonaceous materials were carried out.

2. Graphitization has been defined as the process of thermal recrystallization, and the theoretical foundations of the phenomenon were discussed.

3. As a measure of the propensity towards recrystallization was taken the concentration of free surface energy:

$$\frac{S\sigma}{V} = \frac{S_1\sigma_1}{V} + \frac{S_2\sigma_2}{V} + \frac{S_3\sigma_3}{V} + \sum \dots = \frac{S_1\sigma_1}{V}$$

4. On the basis of the theoretical considerations and the experimental data there was developed a theory of the determining effect of the dispersion structure /state/ on the ability of carbonaceous materials to be graphitized. Only secondary importance was attributed to the role of inorganic admixtures.

5. Here is given a classification of the dispersion structures of different types of carbonaceous materials:

Flaky - carbon black
thin layer - charcoal,
dense crystalline - retort carbon,
foamy - cokes,
crystalline layer - certain anthracites,
layer with crystallites not highly oriented - the majority of coals
and crypto-crystalline graphites.

6. The ease of thermal recrystallization of carbonaceous materials is determined by the density of their structure and the conditions of contact between the graphite crystallites in them.

7. The properties of the graphitization products of carbonaceous materials depend on the degree of orientation of the crystallites composing them. Thus a greasy, flaky product is yielded only by those anthracites which have a complete orientation of the crystallites in the basic plane. In general the greasiness of artificial graphite depends on the degree of development of the basic crystal faces.

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Table 1

Data for Tar Coke

Temperature of Heating in °C	Duration of Heating in Minutes		Specific Resistance in $\rho \cdot 10^4 \text{ ohm} \cdot \text{cm}$	Density	Ash Content in %	Temperature at which 3% is burned in 10 minutes
	Period of Heating Up	Period of Constant Temperature				
Original (crude) Material			100,000	1.620	2.02	440
1,560	60	30	344		2.03	550
1,710	60	30	403		1.99	600
1,710	70	60	304		1.58	610
1,710	50	120	298		1.95	590
1,800	55	30	105		1.59	600
1,850	60	60	108	2.135	1.10	635
1,950	65	30	125		0.75	660
2,000	60	60	119		0.67	640
2,000	50	120	135		0.51	655
2,100	70	30	107		0.18	640-660
2,350	50	30	85		0.18	660
2,350	65	30	112		0.30	630
2,350	60	30	100		0.20	655
2,350	60	120	86	2.166	0.18	645
2,700	65	30	93		0.28	655-670

Table 2

Data for Petroleum Coke

Temperature of Heating in °C	Duration of Heating in Minutes		Specific Resistance in $\rho \cdot 10^4 \text{ ohm} \cdot \text{cm}$	Density	Ash Content in %	Temperature at which 3% is burned in 10 minutes
	Period of Heating Up	Period of Constant Temperature				
1,400	10 days		353	1.960	2.74	460-490
1,500	60	30			2.11	540
1,540	90	30	239		2.19	532
1,630	60	20			1.97	555-565
1,710	60	30	225		1.38	621
1,710	40	30	202		1.84	
1,900	70	30	227		1.13	616
2,000	60	30	138		0.13	
2,140	65	30	154		0.24	660
2,225	70	30	89		0.44	
2,350	65	30	124	2.105	0.22	634
2,400	60	30	138		0.14	
2,610	75	30	117		0.14	645

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Table 3
Data for Coal Coke

Temperature of Heating in °C	Duration of Heating in Minutes		Specific Resistance in $\rho \cdot 10^4 \text{ ohm}\cdot\text{cm}$	Ash Content in %	Temperature at which 3% is burned in 10 minutes
	Period of Heating Up	Period of Constant Tempera- ture			
750			573	16.74	480
1900	35	30	515	17.31	595
2130	50	30	182	8.77	620
2180	70	35	103.6	5.50	645
2520	35	15	81.7	0.15	635
2700	45	25		0.22	665
				0.17	

Table 4
Data for Charcoal

Temperature of Heating in °C	Duration of Heating in Minutes		Specific Resistance in $\rho \cdot 10^4 \text{ ohm}\cdot\text{cm}$	Density	Ash Content in %	Temperature at which 3% is burned in 10 minutes
	Period of Heating Up	Period of Constant Tempera- ture				
700		20	2100	1.466	2.15	430
1000	35	30	1024		5.20	425
1140		30	1089		4.28	435
1710	50	25	880		3.36	490
1900	55	30	746		2.26	490
1900		30	726		2.03	510
2130	55	20	573			537
2170	60	10	486		0.12	565
2300	75	30	650		0.45	535
2350	60	30	600	1.620	0.37	580
2350	90	15	634		0.27	565

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Table 5

Data for Retort Carbon

Temperature of Heating in °C	Duration of Heating in Minutes		Specific Resistance in $\rho \cdot 10^4 \text{ ohm}\cdot\text{cm}$	Ash Content in %	Temperature at which 3% is burned in 10 minutes
	Period of Heating Up	Period of Constant Tempera- ture			
1400	10 days		410	2.86	530
1540	35	30	336	2.81	570
1710	60	30		2.83	620
1805	55	30	300		628
1900	60	30	283	2.05	630
2170	60	30	200		652
2215	60	30	163	0.78	669
2315	55	30	136		678
2570	75	30	134	0.10	665

Table 6

Data for Flame Carbon Black

Temperature of Heating in °C	Duration of Heating in Minutes		Specific Resistance in $\rho \cdot 10^4 \text{ ohm}\cdot\text{cm}$	Density	Temperature at which 3% is burned in 10 minutes
	Period of Heating Up	Period of Constant Tempera- ture			
1000			2500	1.641	475
1490	55	30	860		560
1710	50	30	940		560
1950	55	25	970		560
1950	55	50	1180		590
1950	65	120	1230		540
1990	50	25	1270		
2080	60	30	1230		
2350	60	30	1270	2.002	555
2350	70	35	1260		550
2350	60	35			560
2660	60	30			

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Table 7

Data for Donets Anthracite

Temperature of Heating in °C	Duration of Heating in Minutes		Specific Resistance in $\rho \cdot 10^4 \text{ ohm} \cdot \text{cm}$	Ash Content in %	Volatile	Temperature at which 3% is burned in 10 minutes
	Period of Heating Up	Period of Constant Temperature				
Crude			11 million	6.80	6.80	450
800		20	4200		1.05	450
1000			1236	8.39		460
1400	35	20	648	6.75		510
1710	50	25	495	6.48		516
1990	65	25	405	4.96		610
2130	60	20	253	1.00		640
2225	60	30	239	1.29		655
2350	55	30	236	0.38	0.12	660

Table 8

Data for Poltavka Anthracite

Temperature of Heating in °C	Duration of Heating in Minutes		Specific Resistance in $\rho \cdot 10^4 \text{ ohm} \cdot \text{cm}$	Ash Content in %	Temperature at which 3% is burned in 10 minutes
	Period of Heating Up	Period of Constant Temperature			
Crude			1320	8.42	518
700		20	1220		520
1350	30	20	902	13.94	530
1760	50	25	650	12.53	565
2080	65	20	304	5.59	638
2225	65	30	205	0.53	648
2520	60	30	162	0.12	660

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Table 9

Effect of Admixtures on Graphitization of Petroleum Coke

Admixture	Temperature of Heating in °C	Duration of Heating in Minutes		Specific Resistance in $\rho \cdot 10^4 \text{ ohm} \cdot \text{cm}$	Ash Content in %	Temperature at which 3% is burned in 10 minutes
		Period of Heating Up	Period of Constant Temperature			
CaO	700		20	507	10.67	480
	1140		30	227	5.32	510
	1620		30	239	6.7	510
	2080	45	25	102	0.56	615
	2170		30	81	0.17	615
	2225	50	20	68	0.19	625
	2480	70	30	80	0.02	625
B ₂ O ₃	700			1000	16.61	480
	1900	45	25	207	13.19	630
	1990	55	25	73	11.51	630
	2225	55	25	70	6.18	620
	2580	70	25	68	0.20	660
Fe ₂ O ₃	750			890	20.50	460
	1140		30	291	18.40	510
	1900		30	218	18.00	575
	2040	45	30	144	14.11	645
	2080		30	141	8.50	620
	2220	45	35	123	8.83	620
	2580	70	30	82	0.69	660
Al ₂ O ₃	700		20	997	19.18	460
	1440		30	375	18.50	510
	1620		30	389	18.50	555
	2170	70	30	108	4.16	620
	2200		30	85	0.10	650
SiO ₂	--	--	--	634		
	750	--	--	582	11.68	485
	1760	40	30	246	11.36	510
	1900	45	30	194	11.01	600
	2080	40	30	163	5.74	630
	2450	60	25	99	0.93	660

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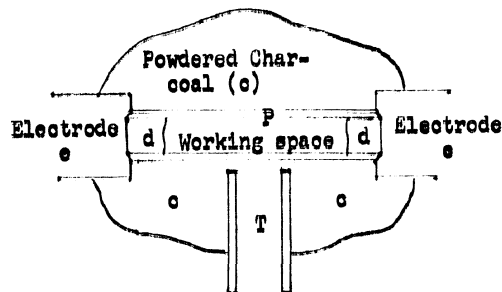


Figure 1. Diagram of furnace for graphitization.

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Figure 2.
Debye-Scherrer x-ray diagram of highly dispersed carbon.

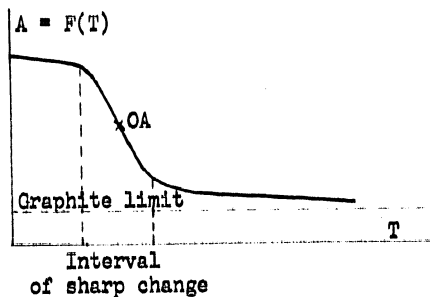


Figure 4. General form of dependency of active properties of the surface (A) on temperature (T).

Figure 3.
Debye-Scherrer x-ray diagram of a graphitic substance with oriented arrangement of crystals.

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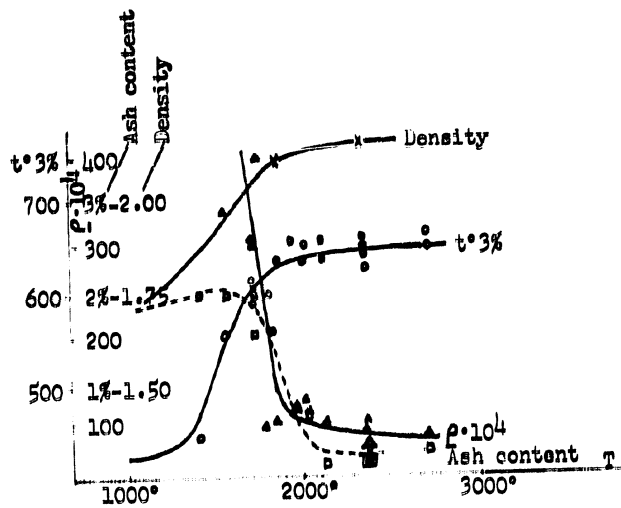


Figure 5. Curves constructed from data for graphitization of Tar Coke.

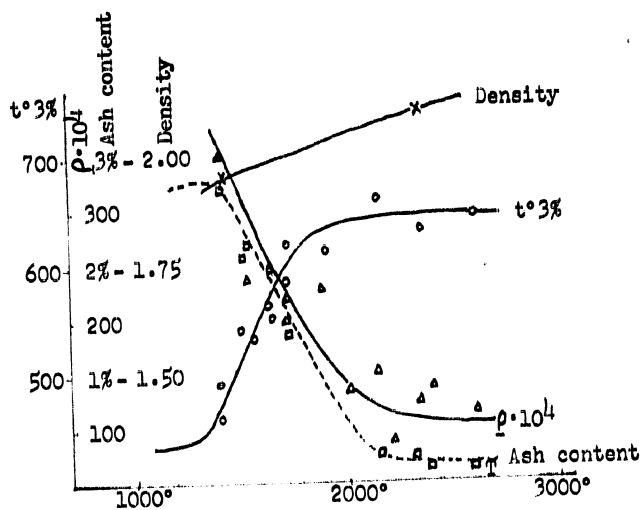


Figure 6. Curves constructed from data for graphitization of Petroleum Coke.

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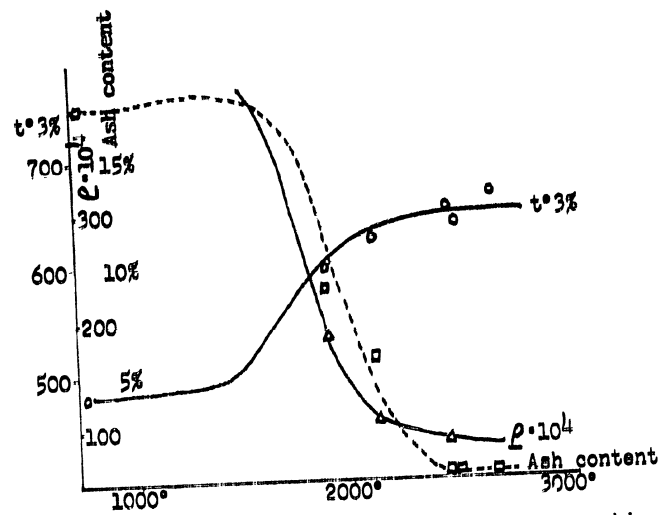


Figure 7. Curves constructed from data for graphitization of Coal Coke.

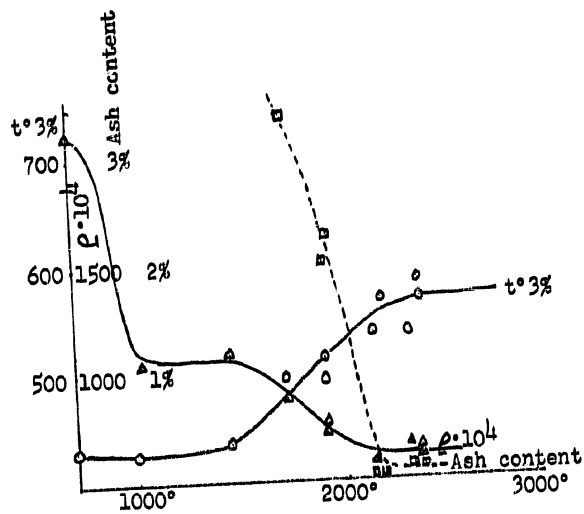


Figure 8. Curves constructed from data for graphitization of Charcoal.

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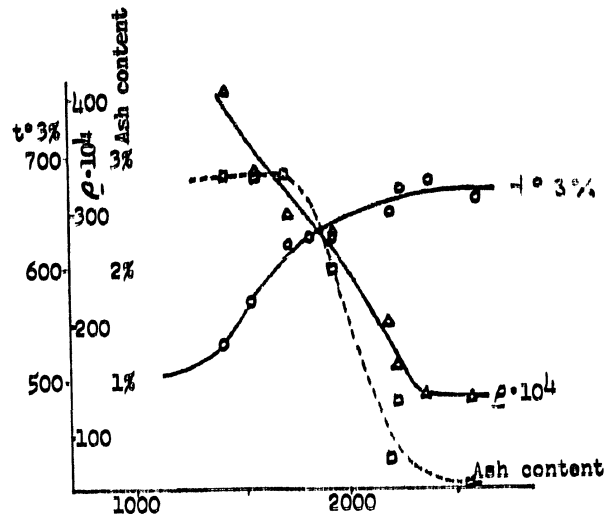
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Figure 9. Curves constructed from data for graphitization of Retort Carbon.

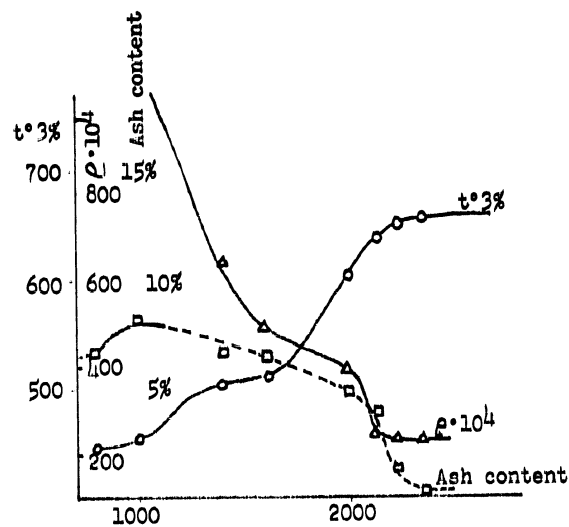


Figure 10. Curves constructed from data for graphitization of Donets anthracite.

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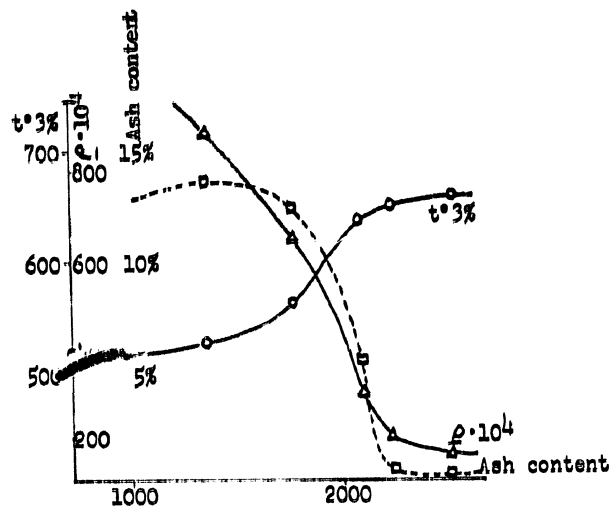
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Figure 11. Curves constructed from curves for graphitization of Poltavka anthracite.

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Figure 12. Drops of fused graphite.

Figure 13. Crystals of almost pure graphite formed after evaporation of iron.

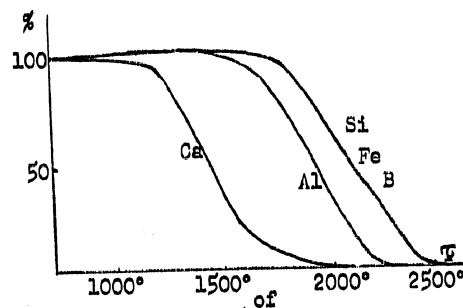


Figure 14. Percent/evaporation of admixtures at various temperatures.

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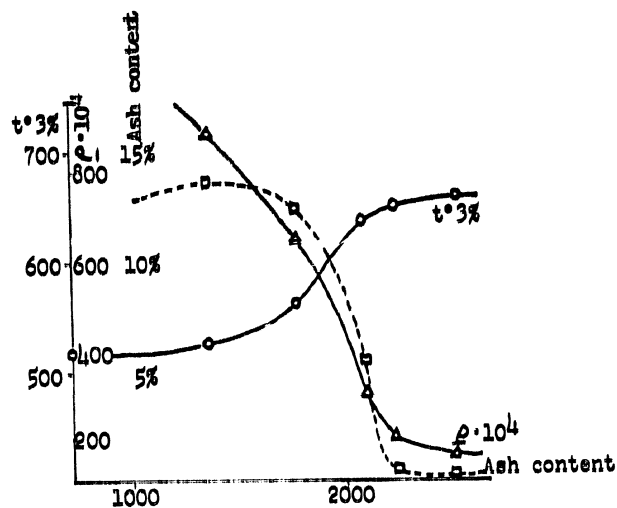
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Figure 11. Curves constructed from curves for graphitization of Poltavka anthracite.

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Figure 12. Drops of fused graphite.

Figure 13. Crystals of almost pure graphite formed after evaporation of iron.

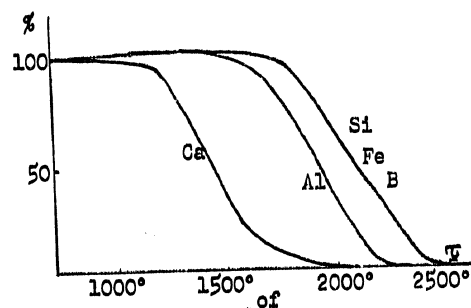


Figure 14. Percent evaporation of admixtures at various temperatures.

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